



(11) Publication number : **0 526 994 A1**

(12)

EUROPEAN PATENT APPLICATION

(21) Application number : **92306458.8**

(51) Int. Cl.⁵ : **C03C 17/23, C03C 17/34**

(22) Date of filing : **14.07.92**

(30) Priority : **18.07.91 US 732018**

(43) Date of publication of application :
10.02.93 Bulletin 93/06

(84) Designated Contracting States :
DE FR GB

(71) Applicant : **FORD MOTOR COMPANY LIMITED**
Eagle Way
Brentwood Essex (GB)
(84) **GB**

(71) Applicant : **FORD FRANCE S. A.**
B.P. 307
F-92506 Rueil-Malmaison Cédex (FR)
(84) **FR**

(71) Applicant : **FORD-WERKE**
AKTIENGESELLSCHAFT
Werk Köln-Niehl Henry-Ford-Strasse Postfach
60 40 02
W-5000 Köln 60 (DE)
(84) **DE**

(72) Inventor : **Brown, Franklin Ibanimina**
300 Riverfront Park, Suite 25C
Detroit, Michigan 48226 (US)
Inventor : **Schulz, Stephen C.**
107 Panorama Drive
Benicia, California 94510 (US)

(74) Representative : **Messulam, Alec Moses**
A. Messulam & Co. 24 Broadway
Leigh on Sea Essex SS9 1BN (GB)

(54) A method of improving the pyrolytic deposition rate of metal oxide film on a glass surface.

(57) A pyrolytic deposition process, for pyrolytic deposition of powder to form metal oxide film, is improved. A substrate is heated, and powder is sprayed at the substrate, using oxygen gas as the carrier gas. Up to one-third greater deposition rate results, as compared to the process using compressed air as the carrier gas. For example organo-cupric powder is sprayed to form a copper oxide film.

EP 0 526 994 A1

This invention relates to a pyrolytic deposition process, and more particularly, to pyrolytic deposition of organo-cupric powder for the formation of copper oxide film.

In the formation of electrochromic devices, and in other applications, copper oxide films are employed, and are deposited on glass substrates. U.S. Patent No. 4,830,471 discloses copper oxide in electrochromic devices, and the content of this patent is incorporated by reference.

It is known to deposit copper oxide film by pyrolytic process. In such a process, a substrate is heated. Cupric acetylacetonate powder is then sprayed on the hot substrate. The cupric acetylacetonate is atomised by compressed air. A film of copper oxide results.

It is an object of this invention to improve the known pyrolytic process by which a copper oxide film is deposited on a substrate such as glass.

An advantage of the invention is that in conditions of actual use, the invention has resulted in a one-third greater deposition rate than the deposition rate of the known process.

Another advantage of the invention is that desirably increased deposition rate may be achieved with minimal change to circumstances of the known process.

This invention is directed to a new pyrolytic deposition process. In particular, this invention is directed to a new pyrolytic process for forming a metal oxide coating on a hot glass substrate.

According to the invention, a metal oxide coating is formed on a hot glass substrate by pyrolysis of a powder at a surface of the substrate. The process of the invention comprises spraying the surface of the substrate with a suspension of powder in oxygen gas.

Examples follow of the practice of the prior art, and of the invention, by the inventors.

A substrate, 12" x 12" x 1/8", of low E glass (glass with pyrolytic SnO₂.F coating) was heated in a vertical furnace to a temperature of 1080°F. It is believed temperatures in the range of approximately 900°F to approximately 1135°F will provide desirable results. Upon exiting the furnace to room temperature, 5g of cupric acetylacetonate powder was sprayed on the hot glass/SnO₂.F surface with a Binks model 171, hand-held floccing gun. The distance from the glass/SnO₂.F surface to the gun nozzle was approximately 5 inches. Compressed air was used for atomising the cupric acetylacetonate powder. The atomisation pressure was 50 psi. Each cycle of heating and spraying is called one application. The Cu_xO film was made in two applications. The film had a thickness of about 600 Å.

The same applications of Cu_xO film were repeated. However dry oxygen gas was used for atomising the cupric acetylacetonate powder. The dry oxygen gas was, as preferred, 99.999% dry. Process variables and settings were otherwise the same. The

Cu_xO film deposited on the glass/SnO₂.F surface had a thickness of about 800 Å.

Oxygen gas as the atomisation agent resulted in a thicker Cu_xO film. The film was thicker by one-third. It is believed that the use of oxygen gas in the manner described above improved the pyrolytic deposition rate of the Cu_xO film by one-third. It is also believed that ozone gas may provide similar results.

Claims

1. A pyrolytic deposition process for forming a metal oxide on a hot glass substrate by pyrolysis of a powder at a surface of said substrate, said process comprising spraying said surface with a suspension of said powder in oxygen gas.
2. A pyrolytic deposition process for forming a copper oxide on a hot glass substrate by pyrolysis of cupric acetylacetonate powder at a surface of said substrate, said process comprising spraying said surface with a suspension of said powder in a carrier gas consisting essentially of dry oxygen gas.
3. A pyrolytic deposition process according to claim 2, wherein said surface comprises a fluorine doped tin oxide coating on said hot glass substrate.



**European Patent
Office**

EUROPEAN SEARCH REPORT

Application Number

EP 92 30 6458

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CL.5)
X	EP-A-0 159 816 (PILKINGTON BROTHERS P.L.C.) * page 5, line 21 - page 6, line 2; claims *	1	C03C17/23 C03C17/34
Y	----	2,3	
Y	EP-A-0 363 044 (FORD MOTOR COMPANY) * page 3, line 7 - page 6, line 53 *	2,3	
A	THIN SOLID FILMS. vol. 185, no. 1, February 1990, LAUSANNE CH pages 123 - 136 O.B.AJAYI ET AL. * page 131, line 6 - page 135 * -----	1-3	
			TECHNICAL FIELDS SEARCHED (Int. CL.5)
			C03C B05D
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 16 OCTOBER 1992	Examiner VAN BOMMEL L.
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- * : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			